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(19) (CA) **APPLICATION FOR CANADIAN PATENT** (12)

(54) A Process for the Production of Epoxide Ring Opening Products

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(72) Daute, Peter - Germany (Federal Republic of) ;
Klein, Johann - Germany (Federal Republic of) ;
Gruetzmacher, Roland - Germany (Federal Republic of) ;
Hoefer, Rainer - Germany (Federal Republic of) ;

(71) Henkel Kommanditgesellschaft auf Aktien - Germany (Federal Republic of) ;

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(71) Anmelder *(für alle Bestimmungsstaaten ausser US):* HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN [DE/DE]; Henkelstraße 67, D-4000 Düsseldorf 13 (DE).

(72) Erfinder; und
(75) Erfinder/Anmelder *(nur für US)* **:** DAUTE, Peter [DE/DE]; Kreuzeskirchstraße 5, D-4300 Essen 1 (DE). KLEIN, Johann [DE/DE]; Beethovenstraße 1, D-4000 Düsseldorf 1 (DE). GRÜTZMACHER, Roland [DE/DE]; Zur Krakau 12, D-5603 Wülfrath (DE). HÖFER, Rainer [DE/DE]; KleverStraße 31, D-4000 Düsseldorf 30 (DE).

(74) Gemeinsamer Vertreter: HENKEL KOMMANDITGESELLSCHAFT AUF AKTIEN; TFP/Patentabteilung, Postfach 10 11 00, D-4000 Düsseldorf 1 (DE).

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(54) Title: METHOD OF PRODUCING EPOXY-RING OPENING PRODUCTS

(54) Bezeichnung: VERFAHREN ZUR HERSTELLUNG VON EPOXIDRINGÖFFNUNGSPRODUKTEN

(57) Abstract

Epoxy-ring opening products may be produced by a) reacting epoxy compounds with nucleophilic agents in the presence of lithium salts and b) subjecting the hydroxy compounds thus formed to transesterification reaction, optionally using fatty-acid glyderide esters.

(57) Zusammenfassung

Epoxidringöffnungsprodukte lassen sich herstellen, indem man a) Epoxidverbindungen mit Nucleophilen in Gegenwart von Lithiumsalzen umsetzt und b) die entstehenden Hydroxyverbindungen gegebenenfalls mit Fettsäureglyceridestern einer Umesterung unterwirft.

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A process for the production of epoxide ring opening products

Field of the Invention

This invention relates to epoxide ring opening products obtainable by reaction of epoxide compounds with nucleophiles in the presence of lithium salts and optionally transesterification of the hydroxy compounds formed as intermediate products with fatty acid glyceride esters, to a process for their production and to their use for the production of polymers.

Prior Art

To produce polyurethane foams, diisocyanates are reacted with substances which contain at least two free hydroxyl groups. Hydroxy compounds particularly suitable for the production of such plastics are normally produced by ring opening of epoxide compounds with nucleophiles, for example alcohols, in the presence of alkaline or acidic catalysts (**Fette, Seifen, Anstrichmitt., 89, 147 (1987)**).

US 4,057,589 for example describes a process for the production of tetrols, in which unsaturated diols are reacted with peracetic acid and the epoxides formed are subsequently hydrolyzed at temperatures of at least 120°C.

EP 0 127 810 A1 describes the sulfuric-acid-catalyzed ring opening of epoxides of unsaturated fatty acid esters with alcohols and their subsequent saponification.

DE 32 46 612 A1 describes a process for the production of modified triglycerides in which epoxidized fats

or oils are reacted with monohydric or polyhydric alcohols in the presence or sulfuric acid, phosphoric acid or sulfonic acids.

Finally, EP 0 257 332 B1 describes a process for the continuous production of 1,2-diols, in which epoxides are subjected to pressure hydrolysis with water in the presence of acidic catalysts.

All these known processes are attended by the disadvantage that the acidic or alkaline catalysts have to be neutralized after the reaction. The salts accumulating have to be removed with considerable effort because otherwise they can cause clouding of the products or may lead to an undesirably violent reaction between polyol and diisocyanate.

Accordingly, the problem addressed by the present invention was to provide a process for the production of epoxide ring opening products which would be free from the disadvantages described above.

Description of the Invention

The present invention relates to a process for the production of epoxide ring opening products, characterized in that

a) epoxide compounds are reacted with nucleophiles in the presence of lithium salts and

b) the hydroxy compounds formed are optionally transesterified with fatty acid glyceride esters.

It has surprisingly been found that the ring opening of epoxides takes place quickly and substantially quantitatively even in the presence of very small quantities of lithium hydroxide and/or lithium fatty acid salts. In

addition, the quantities of lithium still present in the epoxide ring opening products are sufficient to catalyze any transesterification with fatty acid glyceride esters. Another advantage of the process according to the invention is that there is no need for neutralization and/or removal of the lithium salts because the small quantities used do not adversely affect either the properties of the products or their behavior in subsequent processing steps.

Epoxide compounds are known substances and may be obtained in known manner by epoxidation of unsaturated starting materials. Examples of relevant methods are the reaction of olefins with peracetic acid in the presence of acidic catalysts (DE 857 364) or with performic acid formed in situ from formic acid and hydrogen peroxide (US 2,485,160). If the process according to the invention is to be successfully carried out, a substantial percentage content, for example 2 to 40% by weight and preferably 4 to 8.5% by weight, of epoxide oxygen must be present in the epoxide compounds. This includes the observation that not only completely epoxidized, but also partly epoxidized substances may be used in the process according to the invention.

Epoxide compounds in the context of the invention are

a1) ethylene oxide, propylene oxide and/or butylene oxide,

a2) epoxides of olefins corresponding to formula (I):

$$R^1\text{-}CH\text{=}CH\text{-}R^2 \qquad (I)$$

in which R^1 is a linear or branched aliphatic hydrocarbon radical containing 1 to 18 carbon atoms and

R^2 is hydrogen or a linear or branched hydrocarbon radical containing 1 to 8 carbon atoms. Typical examples are the epoxides of oct-1-ene, dec-1-ene, dodec-1-ene, tetradec-1-ene, octadec-1-ene or octadec-9-ene. Epoxides of olefins corresponding to formula (I), in which the sum total of R^1 and R^2 is a number of 8 to 16, are preferred.

a3) **Epoxides of esters** corresponding to formula (II):

$$R^3CO\text{-}OR^4 \qquad \text{(II)}$$

in which R^3CO is an aliphatic acyl radical containing 16 to 24 carbon atoms and 1 to 5 double bonds and R^4 is a linear or branched alkyl radical containing 1 to 4 carbon atoms. Typical examples are the epoxides of palmitoleic acid methyl ester, oleic acid methyl ester, elaidic acid methyl ester, petroselic acid methyl ester, linoleic acid methyl ester or erucic acid methyl ester. Epoxides of esters corresponding to formula (II), in which R^3CO is an aliphatic hydrocarbon radical containing 18 to 22 carbon atoms and 1 or 2 double bonds and R^4 is a methyl group, are preferred.

a4) **Epoxides of esters** corresponding to formula (III):

$$R^5CO\text{-}OR^6 \qquad \text{(III)}$$

in which R^5CO is an aliphatic acyl radical containing 1 to 24 carbon atoms and 0 or 1 to 5 double bonds and R^6 is a linear or branched aliphatic hydrocarbon radical containing 16 to 24 carbon atoms and 1 to 5 double bonds. Typical examples are epoxides of acetic acid oleyl ester, oleic acid oleyl ester or

erucic acid oleyl ester. Epoxides of esters corresponding to formula **(III)**, in which R^5CO is an aliphatic acyl radical containing 18 to 22 carbon atoms and 1 or 2 double bonds and R^6 is an aliphatic hydrocarbon radical containing 16 to 22 carbon atoms and 1 or 2 double bonds, are preferred.

a5) **Epoxides of fatty acid glyceride esters** corresponding to formula **(IV)**:

$$\begin{array}{l} CH_2O\text{-}CO\text{-}R^7 \\ | \\ CH\text{-}O\text{-}CO\text{-}R^8 \\ | \\ CH_2O\text{-}CO\text{-}R^9 \end{array} \qquad \textbf{(IV)}$$

in which R^7CO is a linear or branched aliphatic acyl radical containing 16 to 24 carbon atoms and 1 to 5 double bonds and R^8CO and R^9CO independently of one another represent a linear or branched aliphatic acyl radical containing 6 to 22 carbon atoms and 0 or 1 to 5 double bonds and mixtures thereof. Typical examples are epoxides of peanut oil, coriander oil, cottonseed oil, olive oil, linseed oil, beef tallow, fish oil or, more particularly, soybean oil. Epoxides of glycerol fatty acid esters corresponding to formula **(IV)**, in which R^7CO, R^8CO and R^9CO independently of one another represent aliphatic acyl radicals containing 18 to 22 carbon atoms and predominantly 1 or 2 double bonds, are preferably used.

The following compounds may be used as the **nucleophiles** required for the ring opening of the epoxide compounds:

b1) **Water**

b2) **Alcohols** corresponding to formula (V):

$$R^{10}OH \qquad (V)$$

in which R^{10} is a linear or branched aliphatic hydrocarbon radical containing 1 to 22 carbon atoms and 0 or 1 to 3 double bonds. Typical examples are methanol, ethanol, 1-propanol, 2-propanol, n-butanol, pentanol, hexanol, octanol, decanol, lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl alcohol, oleyl alcohol, behenyl alcohol or erucyl alcohol. Methanol and ethanol are preferably used.

b3) **Polyhydric alcohols** selected from the group consisting of ethylene glycol, diethylene glycol, polyethylene glycols with number average molecular weights in the range from 300 to 1,500, propane-1,2-diol, propane-1,3-diol, glycerol, oligoglycerols having degrees of condensation of on average 2 to 10, trimethylol propane, pentaerythritol, sorbitol and sorbitan.

b4) **Fatty alcohol polyglycol ethers** corresponding to formula (VI):

$$R^{11}O-(CH_2\overset{\overset{\displaystyle R^{12}}{|}}{C}HO)_nH \qquad (VI)$$

in which R^{11} is a linear or branched aliphatic hydrocarbon radical containing 6 to 22 carbon atoms and 0 or 1 to 3 double bonds, R^{12} is hydrogen or a methyl group and n is a number of 1 to 30. Typical examples are adducts of on average 1 to 30 moles of

ethylene and/or propylene oxide with 1 mole of hexanol, octanol, decanol, lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl alcohol, oleyl alcohol, behenyl alcohol or erucyl alcohol. Fatty alcohol polyglycol ethers corresponding to formula (VI), in which R^{11} is a C_{6-18} alkyl radical, R^{12} is hydrogen and n is a number of 1 to 10, are preferably used.

The epoxide compounds and the nucleophiles may be used in molar ratios of 1:10 to 10:1 and preferably in molar ratios of 1:3 to 3:1.

Besides lithium hydroxide, suitable **lithium salts** include lithium soaps, i.e. salts of lithium with optionally hydroxyfunctionalized fatty acids containing 6 to 22 carbon atoms and 0, 1, 2 or 3 double bonds. Typical examples are the lithium salts of caproic acid, caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, palmitoleic acid, stearic acid, oleic acid, elaidic acid, petroselic acid, linoleic acid, linolenic acid, 12-hydroxystearic acid, ricinoleic acid, arachic acid, gadoleic acid, behenic acid or erucic acid. The lithium salts may be used in the form of an aqueous or alcoholic solution. Because they readily lend themselves to metering, the lithium salts of unsaturated fatty acids, which are liquid in any event, are preferably used. the lithium soaps may be directly added to the reaction mixture. However, they may also be formed in situ, for example from lithium hydroxide and a fatty acid ester.

The lithium salts may be used in quantities of 0.001 to 0.1% by weight, preferably in quantities of 0.002 to 0.05% by weight and more preferably in quantities of 0.005 to 0.01% by weight, based on the starting materials. Since lithium ions are capable of exchange with the alkali metal ions present in glasses, which can lead to

a reduction in the concentration of lithium ions, it is advisable to carry out the process in reactors of steel or similarly inert materials.

The ring opening reaction may be carried out in known manner. It has proved to be of advantage to carry out the reaction at the boiling temperature of the nucleophile used or at a temperature in the range from 100 to 250°C. It is not absolutely essential for the ring opening reaction to be complete. Instead, epoxide ring opening products which still have a defined residual epoxide oxygen content, for example 1 to 3% by weight, may also be produced.

Suitable **fatty acid glyceride esters** with which the ring opening products may optionally be transesterified are triglycerides corresponding to formula **(VII)**:

$$
\begin{array}{l}
CH_2O\text{-}CO\text{-}R^{13} \\
| \\
CH\text{-}O\text{-}CO\text{-}R^{14} \\
| \\
CH_2O\text{-}CO\text{-}R^{15}
\end{array}
\qquad \textbf{(VII)}
$$

in which $R^{13}CO$ is a linear or branched, optionally hydroxy- and/or alkoxy-substituted aliphatic acyl radical containing 16 to 24 carbon atoms and 1 to 5 double bonds and $R^{14}CO$ and $R^{15}CO$ independently of one another represent a linear or branched aliphatic acyl radical containing 6 to 24 carbon atoms and 0 or 1 to 5 double bonds. Typical examples are natural fatty acid glyceride esters of vegetable or animal origin based on rapeseed oil, castor oil, palm oil, soybean oil or coconut oil and also chemically modified triglycerides of the soya polyol type which are obtained by epoxidation of soybean oil and subsequent opening of the oxirane rings with suitable nucleophiles, for example alcohols.

The molar ratio between the ring opening products and fatty acid glyceride esters may also be from 10:1 to 1:10 and is preferably from 3:1 to 1:3. The transesterification may be carried out in known manner at temperatures in the range from 100 to 250°C. As mentioned above, the concentration of lithium remaining in the ring opening product is sufficient to catalyze the transesterification reaction also. Accordingly, there is no need for other catalysts to be added. The transesterification reaction may be carried out completely or partly, for example to between 10 and 90% by weight and, more particularly, to between 20 and 70% by weight, based on the fatty acid glyceride ester.

In one preferred embodiment of the invention, the ring opening and transesterification are carried out in one step rather than successively. If unreacted nucleophile still present in the product adversely affects the performance properties of the ring opening products or interferes with their subsequent processing, it may be removed, for example by distillation. It does not matter whether distillation is carried out after the ring opening reaction or after the transesterification reaction.

The present invention also relates to epoxide ring opening products obtainable by

a) reacting epoxide compounds with nucleophiles in the presence of lithium salts and

b) optionally subjecting the hydroxy compounds formed to transesterification with fatty acid glyceride esters.

Industrial Applications

The epoxide ring opening products obtainable by the process according to the invention are suitable as raw materials for the production of polymers. For example, they may be incorporated in alkyd resins by condensation with polybasic acids, e.g. phthalic anhydride, both by way of the hydroxyl functions and by way of epoxide groups still present in the molecule and represent polyfunctional polycondensation units for reaction with polyisocyanates which are of particular importance for the development of polyurethane foams.

Accordingly, the present invention relates to the use of the epoxide ring opening products according to the invention for the production of polymers in which they may be present in quantities of 1 to 90% by weight and preferably in quantities of 10 to 70% by weight, based on the polymers.

The following Examples are intended to illustrate the invention without limiting it in any way.

Examples

Example 1:

A mixture of

260 g	of soybean oil epoxide, epoxide oxygen content 6.68% by weight corresponding to 1.08 mole of epoxide,
741 g (0.83 mole)	of new rapeseed oil, oleic acid content > 80% by weight and
299 g (3.25 moles)	of glycerol

was introduced into a 2-liter three-necked flask equipped with a stirrer, after which 0.13 g (0.0054 mole) of lithium hydroxide, corresponding to 0.01% by weight

(based on the starting materials), was introduced with stirring. The reaction mixture was heated to 220°C. The course of the ring opening reaction was followed on the basis of the change in the concentration of epoxide oxygen in the reaction mixture. The results are set out in Table 1.

Example 2:

As in Example 1, 260 g of soybean oil epoxide, 741 g of rapeseed oil and 299 g of glycerol were reacted at 240°C in the presence of 0.03 g of lithium hydroxide, corresponding to 0.002% by weight (based on the starting materials). The course of the ring opening reaction was followed on the basis of the change in the concentration of epoxide oxygen in the reaction mixture. The results are set out in Table 1.

Example 3:

As in Example 1, 390 g of soybean oil epoxide, corresponding to 1.6 mole of epoxide, 611 g (0.7 mole) of new rapeseed oil and 299 g of glycerol were reacted at 220 to 240°C in the presence of 0.13 g of lithium hydroxide, corresponding to 0.01% by weight, based on the starting materials. The course of the ring opening reaction was followed on the basis of the change in the concentration of epoxide oxygen in the reaction mixture. The results are set out in Table 1.

Example 4:

As in Example 1, 260 g of epoxystearic acid methyl ester, 910 g (1 mole) of new rapeseed oil and 130 g (1.4 mole) of glycerol were reacted at 220 to 240°C in the presence of 0.13 g of lithium hydroxide, corresponding to 0.01% by weight (based on the starting materials). The course of the ring opening reaction was followed on the basis of the

basis of the change in the concentration of epoxide oxygen in the reaction mixture. The results are set out in Table 1.

Example 5:

520 g of epoxystearic acid methyl ester, 520 g (0.6 mole) of new rapeseed oil and 260 g (2.8 moles) of glycerol were reacted at 220 to 240°C in the presence of 0.13 g of lithium hydroxide, corresponding to 0.01% by weight (based on the starting materials). The course of the ring opening reaction was followed on the basis of the change in the concentration of epoxide oxygen in the reaction mixture. The results are set out in Table 1.

Comparison Example 1:

As in Example 1, 260 g of soybean oil epoxide, 741 g of rapeseed oil and 299 g of glycerol were reacted at 240°C in the presence of 0.26 g of potassium hydroxide, corresponding to 0.02% by weight (based on the starting materials). The course of the ring opening reaction was followed on the basis of the change in the concentration of epoxide oxygen in the reaction mixture. The results are set out in Table 1.

Table 1: Epoxide oxygen contents

Ex.	Epoxide oxygen content (% by weight) after h							
	0	1	2	3	4	5	7	10
1	1.3	1.3	0.8	0.4		0.2		
2	1.3	1.2		0.6		0.3	0.2	
3	2.0	1.5		0.8		0.3	0.1	
4	0.9	0.9				0.5	0.3	0.1
5	1.9		1.1		0.6		0.5	0.2
C1	1.3	1.2		0.6		0.6		

Example 6:

2400 g (26 moles) of glycerol were introduced into a 4-liter steel autoclave and, after the addition of 0.45 g of lithium hydroxide, were heated to 80°C. Any traces of water remaining were removed by evacuation and purging with nitrogen five times. The reaction mixture was then heated to 150°C and 1,500 g (26 moles) of propylene oxide were added in portions so that the pressure in the reactor did not exceed 5 bar. On completion of the reaction (approx. 3 h), the reaction mixture was cooled to 80 to 100°C and evacuated for about 15 minutes to remove traces of unreacted propylene oxide. Approximately 2900 g of glycerol propoxylate were obtained in the form of a clear colorless liquid which had a hydroxyl value of 1,200.

498 g (3.3 moles) of the glycerol propoxylate containing lithium hydroxide and 1,495 g (1.7 moles) of new rapeseed oil were transesterified with stirring for 6 h at 240°C. Approximately 1,950 g of a clear pale yellow liquid with the following characteristic data were

obtained:

Hydroxyl value	:	280
Saponification value	:	142
Acid value	:	0.8
Li content	:	7.5 ppm

New Claims 1 to 21

1. A process for the production of epoxide ring opening products, **characterized in that**

a) epoxide compounds are reacted with nucleophiles in the presence of 0.001 to 0.01% by weight - based on the starting materials - of lithium salts and

b) the hydroxy compounds formed are optionally trans-esterified with fatty acid glyceride esters.

2. A process as claimed in claim 1, **characterized in that** ethylene oxide, propylene oxide and/or butylene oxide is/are used as the epoxide compound.

3. A process as claimed in claim 1, **characterized in that** epoxides of olefins corresponding to formula (I):

$$R^1\text{-CH=CH-}R^2 \qquad (I)$$

in which R^1 is a linear or branched aliphatic hydrocarbon radical containing 1 to 18 carbon atoms and R^2 is hydrogen or a linear or branched hydrocarbon radical containing 1 to 8 carbon atoms,
are used as the epoxide compounds.

4. A process as claimed in claim 1, **characterized in that** epoxides of esters corresponding to formula (II):

$$R^3\text{CO-O}R^4 \qquad (II)$$

in which R^3CO is an aliphatic acyl radical containing 16 to 24 carbon atoms and 1 to 5 double bonds and R^4 is a linear or branched alkyl radical containing 1 to 4 carbon atoms,
are used as the epoxide compounds.

5. A process as claimed in claim 1 **characterized in**

that epoxides of esters corresponding to formula (III):

$R^5CO\text{-}OR^6$ (III)

in which R^5CO is an aliphatic acyl radical containing 1 to 24 carbon atoms and 0 or 1 to 5 double bonds and R^6 is a linear or branched aliphatic hydrocarbon radical containing 16 to 24 carbon atoms and 1 to 5 double bonds,
are used as the epoxide compounds.

6. A process as claimed in claim 1, **characterized in that** epoxides of fatty acid glycerol esters corresponding to formula (IV):

$$\begin{array}{l} CH_2O\text{-}CO\text{-}R^7 \\ | \\ CH\text{-}O\text{-}CO\text{-}R^8 \\ | \\ CH_2O\text{-}CO\text{-}R^9 \end{array} \qquad (IV)$$

in which R^7CO is a linear or branched aliphatic acyl radical containing 16 to 24 carbon atoms and 1 to 5 double bonds and R^8CO and R^9CO independently of one another represent a linear or branched aliphatic acyl radical containing 6 to 24 carbon atoms and 0 or 1 to 5 double bonds and mixtures thereof,
are used as the epoxide compounds.

7. A process as claimed in at least one of claims 1 to 6, **characterized in that** water is used as the nucleophile.

8. A process as claimed in at least one of claims 1 to 6, **characterized in that** alcohols corresponding to formula (V):

$R^{10}OH$ (V)

in which R^{10} is a linear or branched aliphatic hydrocarbon radical containing 1 to 22 carbon atoms and 0 or 1 to 3 double bonds,

are used as the nucleophiles.

9. A process as claimed in at least one of claims 1 to 6, **characterized in that** polyhydric alcohols selected from the group consisting of ethylene glycol, diethylene glycol, polyethylene glycols with molecular weights in the range from 300 to 1,500, propane-1,2-diol, propane-1,3-diol, glycerol, oligoglycerols having degrees of condensation of on average 2 to 10, trimethylol propane, pentaerythritol, sorbitol and sorbitan, are used as the nucleophiles.

10. A process as claimed in at least one of claims 1 to 6, **characterized in that** fatty alcohol polyglycol ethers corresponding to formula (VI):

$$R^{11}O-(CH_2\overset{\overset{\displaystyle R^{12}}{|}}{C}HO)_nH \qquad (VI)$$

in which R^{11} is a linear or branched aliphatic hydrocarbon radical containing 6 to 22 carbon atoms and 0 or 1 to 3 double bonds, R^{12} is hydrogen or a methyl group and n is a number of 1 to 30,

are used as the nucleophiles.

11. A process as claimed in at least one of claims 1 to 10, **characterized in that** the epoxide compounds and the nucleophiles are used in a molar ratio of 1:10 to 10:1.

12. A process as claimed in at least one of claims 1 to 11, **characterized in that** lithium hydroxide is used as the lithium salt.

13. A process as claimed in at least one of claims 1 to 11, **characterized in that** lithium soaps of optionally

hydroxy-substituted fatty acids containing 6 to 22 carbon atoms and 0, 1, 2 or 3 double bonds are used as the lithium salts.

14. A process as claimed in at least one of claims 1 to 13, **characterized in that** the ring opening reaction is carried out at the boiling temperature of the nucleophile used or at a temperature in the range from 100 to 250°C.

15. A process as claimed in at least one of claims 1 to 14, **characterized in that** triglycerides corresponding to formula (VII):

$$\begin{array}{l} CH_2O-CO-R^{13} \\ | \\ CH-O-CO-R^{14} \\ | \\ CH_2O-CO-R^{15} \end{array} \qquad (VII)$$

in which $R^{13}CO$ is a linear or branched, optionally hydroxy-and/or alkoxy-substituted aliphatic acyl radical containing 16 to 24 carbon atoms and 1 to 5 double bonds and $R^{14}CO$ and $R^{15}CO$ independently of one another represent a linear or branched aliphatic acyl radical containing 6 to 24 carbon atoms and 0 or 1 to 5 double bonds, are used as the fatty acid glyceride esters in the transesterification reaction.

16. A process as claimed in at least one of claims 1 to 15, **characterized in that** the epoxides and the fatty acid glyceride esters are used in a molar ratio of 10:1 to 1:10.

17. A process as claimed in at least one of claims 1 to 18, **characterized in that** the transesterification is carried out at a temperature in the range from 100 to 250°C.

18. A process as claimed in at least one of claims 1 to 17, **characterized in that** in that the ring opening and

transesterification reactions are carried out in a single step.

19. Epoxide ring opening products obtainable by

a) reacting epoxide compounds with nucleophiles in the presence of 0.001 to 0.01% by weight - based on the starting materials - of lithium salts and

b) optionally subjecting the hydroxy compounds formed to transesterification with fatty acid glyceride esters.

20. The use of the epoxide ring opening products obtained by the process claimed in claims 1 to 18 for the production of alkyd resins and polyurethane foams.

21. The use of the epoxide ring opening products claimed in claim 19 for the production of alkyd resins and polyurethane foams.